1 **PRODUCTION OF HIGH QUALITY**
2 **LUBRICANT BRIGHT STOCK**
3
4 <u>FIELD OF THE INVENTION</u>
5
6 The present invention relates to a process for producing a high quality
7 lubricant bright stock from heavy petroleum feedstocks.
8
9 <u>BACKGROUND OF THE INVENTION</u>
10
11 This invention is directed to a process for preparing a high quality lubricant
12 bright stock from heavy petroleum feedstocks. These heavy feedstocks are
13 often contaminated with sulfur, nitrogen, asphaltenic and metal contaminants,
14 which must be removed in preparing the lubricant base stock. They also
15 generally contain significant amounts of waxy materials.
16
17 Low valued oils such as deasphalted oil (DAO) are increasingly being
18 hydrotreated and used as FCC cracker feed to produce gasoline. Severity of
19 deasphalting is much less for making fuels than for making lubricant bright
20 stock. Consequently, the purity of fuels-application DAO is too low to make
21 lubricant bright stock with adequate stability for use in finished lubricant
22 applications. However, increasingly stringent mandated limits on gasoline
23 sulfur require higher severity in fuels DAO hydrotreating. These changes
24 improve the quality of DAO as a feed to hydrocracking to produce high
25 viscosity lubricant base oils. Nevertheless, the highest boiling portion of DAO
26 contains high molecular weight waxes which are difficult to remove, leading to
27 low yields of high cloud point products. The highest boiling fractions also
28 contain large polycyclic molecules, which are difficult to completely saturate in
29 hydrofinishing and which lead to stability problems.
30
31 Conventional high quality Group II lubricant neutral oils having excellent
32 oxidation stability, good low temperature properties and high viscosity indices
33 are generally made by hydrocracking gas oils, followed by dewaxing and

optionally mild hydrotreating. Lubricant base stocks having viscosities of up to about 100 cSt, measured at 40°C, are made in this manner. Higher viscosity oils, for example, bright stocks and similar oils with viscosities of 220 cSt or greater, are generally not made by isomerization dewaxing and generally do not posses the high quality of Group II base oils prepared by isomerization dewaxing. High viscosity oils of improved quality are in general demand, especially for non-engine oil applications, such as industrial oils.

Such high viscosity oils generally require some bright stock in their formulation, the amount of which depends on the product. In typical formulations, Group I bright stock is used, which degrades the product when blended with neutral oil. One problem with the quality of bright stock is that it is not a distillate and is typically of low quality, particularly with respect to oxidation stability. Thus, there is a need for a method for producing an oxidation stable, good quality lubricant bright stock.

In addition, feedstocks which are useful for making lubricant bright stock have generally been limited to gas oils, and specifically vacuum gas oils. Residuum streams are generally difficult to process for lubricant base oils. Not only are the sulfur, nitrogen and aromatic contents of residuum streams very high, but the waxy materials present in these residuum streams are difficult to process in the production of low pour point base oil products. It is especially, therefore, to be able to produce good quality lubricant bright stock from residuum-derived streams.

SUMMARY OF THE INVENTION

The present invention relates to a process for producing a lubricant bright stock from a very heavy feed, having a normal boiling end point within the range of 1150°F to 1300°F. The very heavy feed is obtained from petroleum crude. The bright stock produced by the present process has a reduced cloud point and better oxidation stability relative to bright stocks prepared by conventional methods.

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1   The present invention provides a process for producing a stable lubricant
2   bright stock comprising the steps of providing a petroleum residuum-derived
3   stream having a sulfur content of less than 1% and a nitrogen content of less
4   than 0.5%; separating the residuum-derived stream at a distillation cut point in
5   the range of 1150°F to 1300°F, into a heavy fraction and at least one light
6   fraction; hydrocracking the at least one light fraction under lube hydrocracking
7   in a lube hydrocracking zone in the presence of a hydrocracking catalyst and
8   hydrogen under conditions to reduce the concentration of sulfur and nitrogen
9   to suitable levels for hydroisomerization dewaxing; and dewaxing at least a
10 portion of the hydrocracked stream in a hydroisomerization zone in the
11 presence of an isomerization catalyst and hydrogen under hydroisomerization
12 conditions to produce a lubricant bright stock.
13
14 In one embodiment, a vacuum residuum fraction, which is optionally
15 hydrotreated/hydrocracked prior to further treating, is separated in a high
16 temperature fractionation step, at a cut point temperature in the range of
17 1150°F to 1300°F, into at least a heavy fraction and a light fraction. The light
18 fraction is further processed by hydroisomerization dewaxing to prepare a low
19 haze bright stock.
20
21 Conventional methods for preparing bright stock by hydroisomerization
22 dewaxing a vacuum residuum fraction, a hydrocracked vacuum residuum
23 fraction, deasphalted oil or hydrocracked deasphalted oil generally produces a
24 bright stock with unacceptable haze-forming tendencies.
25
26 Among other factors, the present invention is based on the surprising
27 discovery that the high temperature fractionation, with a cut point in the range
28 of 1150°F to 1300°F, concentrates the haze-forming components found in the
29 vacuum residuum fraction in the heavy fraction, and provides a low haze, and
30 relatively low sulfur containing light fraction which can be processed using
31 conventional methods for preparing a bright stock lubricant. The preferred
32 method of fractionating at the high temperatures required to achieve the
33 desired separation is a short path distillation, such as a wiped film evaporator.

DETAILED DESCRIPTION

Feed Stream:

The petroleum feed stream which is treated in the present invention is a residuum fraction, derived from the fractionation of a petroleum feed, preferably a crude feed. Other feeds which may be treated in the present invention include deasphalted oil, heavy coker products, and the like.

Deasphalted oil (DAO) may be recovered from a conventional deasphalting process, such as a solvent deasphalting process. Such processes are well known in the art. A process for preparing a DAO which is useful in the present invention is described, for example, in U.S. Patent No. 6,001,886, which is incorporated by reference. In a deasphalting process, a residuum is subjected to counter-current contacting at solvent deasphalting conditions, generally at a temperature in the range of 50°F to 400°F, preferably 150°F to 300°F, a dosage of from 0.5 to 10, preferably 1.0 vol. to 3.0 vol. solvent/vol. oil and a pressure of atmospheric pressure to 400 psig, preferably atmospheric pressure to 50 psig. The actual deasphalting conditions chosen are dependent on the solvent. That is, the temperature chosen should not exceed the critical temperature of the solvent and the pressure is maintained above the autogenous pressure to prevent vaporization. Deasphalted oil and solvent are removed by distillation or by stripping the asphalt layer, leaving behind a viscous asphaltic residue. Deasphalting solvents which are useful for this purpose include C_2 to C_8 paraffins, furfural and N-methyl-2-pyrrolidone. Propane and butane are preferred. Pentane is the most suitable solvent if high yields of deasphalted oil are desired. These lower-boiling paraffinic solvents may also be used as mixtures with alcohols such as methanol and isopropanol. Propane as a solvent results in the lowest yield of deasphalted oil and highest yield of asphaltic residue. Because propane is the preferred commercial solvent, the process is often referred to as propane deasphalting. Iso-butane and n-butane are also used commercially. The Rose (Residual Oil Solvent Extraction) process has been the object of many patents disclosing

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1   different operating conditions, or the use of several solvents as specified, for

2   example, in U.S. Patent Nos. 3,830,732 and 4,125,459. The preferred solvent

3   in the Rose process is pentane. The Rose process includes a step under

4   supercritical conditions adapted to separate the solvent from the deasphalted

5   oil.

6

7   **Hydrotreating/Hydrocracking the Residuum Feed:**

8

9   The residuum feed stream is optionally upgraded as necessary prior to

10   separation in the deep cut distillation stage. The feed to the deep cut

11   distillation should have a sulfur content of less than 1% and a nitrogen content

12   of less than 0.5%. The method of upgrading depends, at least in part, on the

13   quality of the feedstock and the quality of the desired bright stock lubricant

14   product. For example, the feed stream may desirably be hydrotreated to

15   remove sulfur without extensive molecular weight conversion by

16   hydrocracking. As used here, the upgrading step prior to separation is

17   identified as a hydrotreating/hydrocracking step to indicate the range of

18   upgrading severities which may be used in the present process.

19

20   Hydrotreating refers to a catalytic process, usually carried out in the presence

21   of free hydrogen, in which the primary purpose is the desulfurization and/or

22   denitrification of the feed stock. Generally, in hydrotreating operations,

23   cracking of the hydrocarbon molecules (i.e., breaking the larger hydrocarbon

24   molecules into smaller hydrocarbon molecules) is minimized and the

25   unsaturated hydrocarbons are either fully or partially hydrogenated.

26   Hydrotreating conditions include a reaction temperature between

27   400°F to 900°F (204°C to 482°C), preferably 650°F to 850°F

28   (343°C to 454°C.); a pressure between 500 psig to 5000 psig (pounds per

29   square inch gauge) (3.5 MPa to 34.6 MPa), preferably 1000 psig to 3000 psig

30   (7.0 MPa to 20.8 MPa); a feed rate (LHSV) of 0.5 hr^{-1} to 20 hr^{-1} (v/v); and

31   overall hydrogen consumption 300 to 2000 standard cubic feet per barrel of

32   liquid hydrocarbon feed (53.4-356 m^3 H$_2$/m^3 feed). The hydrotreating catalyst

33   for the beds will typically be a composite of a Group VI metal or compound

- 5 -

1   thereof, and a Group VIII metal or compound thereof supported on a porous

2   refractory base such as alumina and silica-alumina. Examples of

3   hydrotreating catalysts are alumina supported cobalt-molybdenum, nickel

4   sulfide, nickel-tungsten, cobalt-tungsten and nickel-molybdenum. Typically,

5   such hydrotreating catalysts are presulfided.

6

7   Hydrocracking is a process of breaking larger hydrocarbon molecules into

8   smaller ones. It can be affected by contacting the particular fraction or

9   combination of fractions, with hydrogen in the presence of a suitable

10   hydrocracking catalyst. The hydrocracking step reduces the size of the

11   hydrocarbon molecules, hydrogenates olefin bonds, hydrogenates aromatics,

12   opens rings, and removes traces of heteroatoms. Typical hydrocracking

13   conditions include: reaction temperature, 400°F to 950°F (204°C to 510°C),

14   preferably 650°F to 850°F (343°C to 454°C); reaction pressure 500 psig to

15   5000 psig (3.5 MPa to 34.5 MPa), preferably 1500 psig to 3500 psig

16   (10.4 MPa to 24.2 MPa); liquid hourly space velocity (LHSV),

17   0.1 hr^{-1} to 15 hr^{-1} (v/v), preferably 0.25 hr^{-1} to 2.5 hr^{-1}; and hydrogen

18   consumption 500 to 2500 standard cubic feet per barrel of liquid hydrocarbon

19   feed (89.1-445 m^3 H_2/m^3 feed). The hydrocracking catalyst generally

20   comprises a cracking component, a hydrogenation component, and a binder.

21   Such catalysts are well known in the art. The cracking component may

22   include an amorphous silica-alumina phase and/or a zeolite, such as a Y-type

23   or USY zeolite. The binder is generally silica, alumina or silica-alumina. The

24   hydrogenation component will be a Group VI or Group VIII metal or oxides or

25   sulfides thereof, preferably one or more of molybdenum, tungsten, cobalt, or

26   nickel, or the sulfides or oxides thereof. If present in the catalyst, these

27   hydrogenation components generally make up from about 5% to about

28   40% by weight of the catalyst. Alternatively, platinum group metals, especially

29   platinum and/or palladium, may be present as the hydrogenation component,

30   either alone or in combination with the base metal hydrogenation components

31   molybdenum, tungsten, cobalt, or nickel. If present, the platinum group metals

32   will generally make up from about 0.1% to about 2% by weight of the catalyst.

Deep Cut Distillation:

Once a suitable residuum-derived petroleum feedstock has been obtained, it is next separated into a heavy fraction and a light fraction by a deep cut distillation. The deep cut distillation separates the heavy feed stream at a cut point in the range of 1150°F to 1300°F into a heavy fraction and a light fraction, the latter of which undergo further separation by distillation. The cut point is the temperature at which there are equal amounts of material overlapping from adjacent cuts. When data is not available for one or both adjacent cuts, cut point estimates are the 10 and 90 percent points of the distillation curve. The heavy fraction has a boiling point predominantly above the cut point and the at least one light fraction has a boiling point predominantly below the cut point. The heavy fraction may be used as feed to the FCC or recycled to the hydrocracker. At least a portion of the light fraction is used as a feedstock for hydrocracking discussed more fully below.

Special care is required to separate very high boiling materials in order to minimize product degradation. WO 00/11113, which is incorporated herein by reference, describes the use of special packing, stream stripping and high vacuum to achieve high temperature separations without product degradation. Other commercially available methods employ techniques developed as molecular distillation methods. These are described in detail in, for example, G. Burrows, *Molecular Distillation*, Oxford: Clarendon Press, 1960. Such short-path distillation methods include falling film evaporators and wiped film evaporators. An example of short path distillation is described in U.S. Patent No. 4,925,558, incorporated herein by reference.

The American Society for Testing and Materials (ASTM) has established guidelines for simulated distillation analyses, which include samples that have atmospheric equivalent boiling points (AEBP) in the range of about -44°F to 1139°F. These include ASTM Methods D2887 and D3710. ASTM Method D2887 has an upper temperature limit for petroleum products with a final boiling point of 1000°F at atmospheric pressure. For analysis of

1   heavier samples, such as crude oils, HTSD method D6352 extends the AEBP

2   distribution to temperatures upwards of 1300°F to 1380°F. So, the cut point of

3   the deep cut distillation of the present invention should be determined by

4   ASTM method D6352 or an equivalent method. In practice, ASTM method

5   D6352 gives distillation curve data as output and not cut point. Those skilled

6   in the art know that such distillation curve data are primary requisites for cut

7   point determination. Cut points are typically at 10 and 90 percent points on

8   distillation curves. These 10 and 90 percent points are much more reliably

9   measured than the start and end point, because they are much less

10   dependent on details of D6352 execution (such as manufacturer of equipment

11   and selection of computer software). Of course, as noted above, exact cut

12   point determination requires knowledge of adjacent cut distillation curves and

13   relative amounts.

14

15   **Hydrocracking:**

16

17   At least a portion of the light fraction from the deep cut distillation is

18   hydrocracked in a lube hydrocracking zone in the presence of a hydrocracking

19   catalyst and hydrogen under conditions to reduce the concentration of sulfur

20   and nitrogen to suitable levels for hydroisomerization dewaxing. The lube

21   hydrocracking conditions should also be selected to increase the VI to meet

22   VI specifications. Typically the solvent dewaxed VI of the feed should be

23   greater than 90 and preferably greater than 95. The concentration of nitrogen

24   in the feed for hydroisomerization dewaxing should be less than 50 ppm,

25   preferably less than 30 ppm, and more preferably less than 10 ppm. The

26   concentration of sulfur in the feed for hydroisomerization dewaxing should be

27   less than 100 ppm, preferably less than 50 ppm and more preferably less than

28   20 ppm.

29

30   Therefore, hydrocracking conditions for hydrocracking the light fraction

31   recovered from deep cut distillation, as broadly described, corresponds to

32   hydrocracking conditions for hydrocracking the waxy heavy feed stream.

33   Typical hydrocracking conditions include: reaction temperature,

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1   400°F to 950°F (204°C to 510°C), preferably 650°F to 850°F

2   (343°C to 454°C); reaction pressure 500 psig to 5000 psig

3   (3.5 MPa to 34.5 MPa), preferably 1500 psig to 3500 psig

4   (10.4 MPa to 24.2 MPa); liquid hourly space velocity (LHSV),

5   0.1 hr^{-1} to 15 hr^{-1} (v/v), preferably 0.25 hr^{-1} to 2.5 hr^{-1}; and hydrogen

6   consumption 500 to 2500 standard cubic feet per barrel of liquid hydrocarbon

7   feed (89.1-445 m^3 H_2/m^3 feed). The hydrocracking catalyst generally

8   comprises a cracking component, a hydrogenation component, and a binder.

9   Such catalysts are well known in the art. The cracking component may

10 include an amorphous silica-alumina phase and/or a zeolite, such as a Y-type

11 or USY zeolite. The binder is generally silica, alumina or silica-alumina. The

12 hydrogenation component will be a Group VI or Group VIII metal or oxides or

13 sulfides thereof, preferably one or more of molybdenum, tungsten, cobalt, or

14 nickel, or the sulfides or oxides thereof. If present in the catalyst, these

15 hydrogenation components generally make up from about 5% to about 40%

16 by weight of the catalyst. Alternatively, platinum group metals, especially

17 platinum and/or palladium, may be present as the hydrogenation component,

18 either alone or in combination with the base metal hydrogenation components

19 molybdenum, tungsten, cobalt, or nickel. If present, the platinum group metals

20 will generally make up from about 0.1% to about 2% by weight of the catalyst.

21

22 **Hydroisomerization:**

23

24 At least a portion of the effluent from the lube hydrocracking is subjected to

25 hydroisomerization dewaxing to produce a low haze bright stock.

26

27 Hydroisomerization dewaxing catalysts useful in the present invention

28 generally comprise one or more shape selective intermediate pore size

29 molecular sieves and optionally a catalytically active metal hydrogenation

30 component on a refractory oxide support. The shape selective intermediate

31 pore size molecular sieves used alone or in combination in the practice of the

32 present invention are generally 1-D 10-, 11-, or 12-ring molecular sieves. The

33 preferred molecular sieves of the invention are of the 1-D 10-ring variety,

- 9 -

1   where 10-(or 11-or 12-) ring molecular sieves have 10 (or 11 or 12)

2   tetrahedrally-coordinated atoms (T-atoms) joined by oxygens. In the

3   1-D molecular sieve, the 10-ring (or larger) pores are parallel with each other,

4   and do not interconnect. The classification of intrazeolite channels as

5   1-D, 2-D and 3-D is set forth by R.M. Barrer in Zeolites,

6   Science and Technology, edited by F.R. Rodrigues, L.D. Rollman and

7   C. Naccache, NATO ASI Series, 1984 which classification is incorporated in

8   its entirety by reference (see particularly page 75).

9

10   Preferred shape selective intermediate pore size molecular sieves used for

11   hydroisomerization dewaxing are based upon aluminum phosphates, such as

12   SAPO-11, SAPO-31, and SAPO-41. SAPO-11 and SAPO-31 are more

13   preferred, with SAPO-11 being most preferred. SM-3 is a particularly

14   preferred shape selective intermediate pore size SAPO, which has a

15   crystalline structure falling within that of the SAPO-11 molecular sieves. The

16   preparation of SM-3 and its unique characteristics are described in

17   U.S. Patent Nos. 4,943,424 and 5,158,665. Also preferred shape selective

18   intermediate pore size molecular sieves used for hydroisomerization

19   dewaxing are zeolites, such as ZSM-22, ZSM-23, ZSM-35, ZSM-48, ZSM-57,

20   SSZ-32, offretite, and ferrierite. SSZ-32 and ZSM-23 are more preferred.

21

22   A particularly preferred intermediate pore size molecular sieve, which is useful

23   in the present process, is described, for example, in

24   U.S. Patent Nos. 5,135,638 and 5,282,958, the contents of which are

25   hereby incorporated by reference in their entirety. In

26   U.S. Patent No. 5,282,958, such an intermediate pore size molecular

27   sieve has a crystallite size of no more than about 0.5 microns and pores

28   with a minimum diameter of at least about 4.8 Å and with a maximum

29   diameter of about 7.1 Å. The catalyst has sufficient acidity so that 0.5 grams

30   thereof when positioned in a tube reactor converts at least 50% of

31   hexadecane at 370°C, a pressure of 1200 psig, a hydrogen flow of

32   160 ml/min, and a feed rate of 1 ml/hr. The catalyst also exhibits isomerization

33   selectivity of 40 or greater (isomerization selectivity is determined as follows:

1   100 x (weight % branched C_{16} in product) / (weight % branched C_{16} in product

2   + weight % C_{13-} in product) when used under conditions leading to

3   96% conversion of normal hexadecane (n-C_{16}) to other species.

4

5   Such a particularly preferred molecular sieve may further be characterized by

6   pores or channels having a crystallographic free diameter in the range of from

7   about 4.0 Å to about 7.1 Å , and preferably in the range of 4.0 Å to 6.5 Å., The

8   crystallographic free diameters of the channels of molecular sieves are

9   published in the "Atlas of Zeolite Framework Types", Fifth Revised Edition,

10   2001, by Ch. Baerlocher, W.M. Meier, and D.H. Olson, Elsevier, pages 10-15,

11   which is incorporated herein by reference.

12

13   If the crystallographic free diameters of the channels of a molecular sieve are

14   unknown, the effective pore size of the molecular sieve can be measured

15   using standard adsorption techniques and hydrocarbonaceous compounds of

16   known minimum kinetic diameters. See Breck, Zeolite Molecular Sieves, 1974

17   (especially Chapter 8); Anderson et al., J. Catalysis 58, 114 (1979); and

18   U.S. Patent No. 4,440,871, which is incorporated herein by reference. In

19   performing adsorption measurements to determine pore size, standard

20   techniques are used. It is convenient to consider a particular molecule as

21   excluded if does not reach at least 95% of its equilibrium adsorption value on

22   the molecular sieve in less than about 10 minutes (p/po=0.5; 25°C).

23   Intermediate pore size molecular sieves will typically admit molecules having

24   kinetic diameters of 5.3Å to 6.5 Å with little hindrance.

25

26   Hydroisomerization dewaxing catalysts useful in the present invention

27   optionally comprise a catalytically active hydrogenation metal. Typical

28   catalytically active hydrogenation metals used alone or in combination include

29   chromium, molybdenum, nickel, vanadium, cobalt, tungsten, zinc, platinum,

30   and palladium. The metals platinum and palladium are especially preferred,

31   with platinum most especially preferred. If platinum and/or palladium is used,

32   the total amount of active hydrogenation metal is typically in the range of

1 0.1 to 5 weight % of the total catalyst, usually from 0.1 to 2 weight %, and not

2 to exceed 10 weight %.

3

4 The refractory oxide support may be selected from those oxide supports

5 which are conventionally used for catalysts, including silica, alumina,

6 silica-alumina, magnesia, titania and combinations thereof.

7

8 The catalytic hydroisomerization conditions employed depend on the feed

9 used for the hydroisomerization and the desired pour point of the product.

10 Generally, the temperature is from about 200°C to about 475°C, preferably

11 from about 250°C to about 450°C. The pressure is typically from about

12 15 psig to about 3000 psig, preferably from about 50 psig to about 2500 psig,

13 more preferably from about 100 psig to about 1000 psig, and most preferably

14 from about 150 psig to about 600 psig. The liquid hourly space

15 velocity (LHSV) is preferably from about 0.1 hr^{-1} to about 20 hr^{-1}, more

16 preferably from about 0.1 hr^{-1} to about 5 hr^{-1}, and most preferably from about

17 0.1 hr^{-1} to about 1.0 hr^{-1}. Low pressure and low liquid hourly space velocity

18 provide enhanced isomerization selectivity, which results in more

19 isomerization and less cracking of the feed, thus producing an increased

20 yield.

21

22 Hydrogen is preferably present in the reaction zone during the catalytic

23 isomerization process. The hydrogen to feed ratio is typically from about

24 500 SCF/bbl to about 30,000 SCF/bbl (standard cubic feet per barrel),

25 preferably from about 1000 SCF/bbl to about 20,000 SCF/bbl.

26

27 **Hydrofinishing:**

28

29 The product from the hydroisomerization step may optionally be hydrofinished

30 in order to stabilize the lubricant product by reducing olefins and aromatics.

31 Hydrofinishing is typically conducted at temperatures ranging from about

32 300°F to about 600°F, at pressures from about 400 psig to about 3000 psig, at

33 space velocities (LHSV) from about 0.1 to about 20, and hydrogen recycle

1 rates of from about 400 SCF/bbl to about 1,500 SCF/bbl. The hydrogenation
2 catalyst employed must be active enough not only to hydrogenate the olefins,
3 diolefins and color bodies within the lube oil fractions, but also to reduce the
4 aromatic content (color bodies). The hydrofinishing step is beneficial in
5 preparing an acceptably stable lubricating oil. Suitable hydrogenation
6 catalysts include conventional metallic hydrogenation catalysts, particularly
7 the Group VIII metals such as cobalt, nickel, palladium and platinum. The
8 metals are typically associated with carriers such as bauxite, alumina, silica
9 gel, silica-alumina composites, and crystalline aluminosilicate zeolites.
10 Palladium is a particularly preferred hydrogenation metal. If desired,
11 non-noble Group VIII metals can be used with molybdates. Metal oxides or
12 sulfides can be used. Suitable catalysts are disclosed in
13 U.S. Patent Nos. 3,852,207; 4,157,294; 3,904,513 and 4,673,487, which are
14 incorporated by reference herein.
15
16 Additionally, U.S. Patent No. 6,337,010, which is incorporated by reference
17 herein discloses a process scheme for producing lubricating base oil with low
18 pressure dewaxing and high pressure hydrofinishing and discloses operating
19 conditions for lube hydrocracking, isomerization and hydrofinishing that may
20 be useful in practicing the present invention.
21
22 **Clay Treating:**
23
24 The low haze heavy base oil may optionally also be treated by a clay treating
25 step, either following hydroisomerization or following hydrofinishing to remove
26 any remaining traces of haze or haze precursors. A suitable clay treating
27 process is described, for example, in U.S. Patent No. 6,468,418, which is
28 incorporated by reference.
29
30 The following example is intended to illustrate the present invention and is not
31 intended to limit the invention in any way.

1   <u>EXAMPLE</u>

2

3   The following example describes a method of the invention for preparing low

4   haze bright stock. An Alaska North Slope/Arabian Light/ Arabian Medium

5   crude blend was fractionated in an atmospheric/vacuum distillation and the

6   vacuum column bottoms upgraded by solvent deasphalting and the DAO

7   hydrocracked. The de-asphalting process and the hydrocracking process

8   were conventional.

9

10   The residuum (i.e., bottoms) fraction from an atmospheric fractionation of the

11   hydrocracked DAO was topped by vacuum distillation at 700°F, and

12   the residuum fraction separated by wiped film evaporator distillation at a

13   1200°F cut point. The 1200°F+ bottoms portion was 13.3 weight % of the

14   700°F+ fraction.

15

16   The 700°F to 1200°F distillate had the following properties:

17

18   API Gravity   20.7
19   Nitrogen, ppm   988
20   Sulfur, ppm   2227
21   Viscosity, cSt, 100°C   21.55
22   Viscosity Index   70
23   Sim. Dist., weight %, °F, D6352
24   10%   767
25   50%   977
26   90%   1158
27

This feed was hydrocracked using a conventional commercial Ni-W-SiO2-Al2O3 hydrocracking catalyst at 720/732°F reaction temperature (2 catalyst zones) at 2052 psig pressure (1968 psia H_2 pressure), 0.25 hr^{-1} feed rate, and 5200 SCFB recycle H_2 rate, and the hydrocracker effluent separated first in conventional flash separation zones and then by atmospheric/vacuum distillation. The bright stock (i.e., bottoms) fraction from the vacuum distillation was at a yield of 22.7 vol% and had the following properties:

API Gravity	27.1	
Nitrogen, ppm	7	
Sulfur, ppm	14	
Viscosity, cSt, 100°C	25.67	
Viscosity Index	97	
Wax, weight %	16.3	
Sim. Dist., weight %, °F, D6352		
	10%	943
	30%	1026
	50%	1070
	70%	1117
	90%	1182

The bright stock was converted by isomerization dewaxing at 610°F reaction temperature at 1950 psig pressure (1878 psia H_2 pressure), 1.3 hr^{-1} feed rate, and 3000 SCFB once-through H_2 rate, over a Pt/SSZ-32 catalyst (containing 35% alumina binder), followed by hydrofinishing at 450°F, 1950 psig pressure, 1.0 hr^{-1} feed rate, and 3000 SCFB once-through H_2 rate, over a Pt-Pd/SiO$_2$-Al$_2$O$_3$ catalyst, into a low haze bright stock lubricant product having the following properties:

API Gravity	27.0	
Viscosity, cSt		
	(measured at 40°C)	355.7
	(measured at 100°C)	25.68
Viscosity Index	95	
Pour Point, °C	-20°	
Cloud Point, °C	+2°	
Oxidator BN, hrs	21.5	
Distillation by D6352, weight %, °F		
	10%	956
	30%	1009
	50%	1054
	70%	1090
	90%	1135

1   Lube yield was 94.7 weight %. Conducting the same set of reaction steps,

2   using a full bottoms fraction from the hydrocracked DAO would be expected to

3   produce an isomerization dewaxing/hydrofinishing product having a cloud

4   point of at least 15°C.

5

6   By contrast, solvent dewaxing the same bright stock feed produced an oil with

7   a pour point of -21°C but a VI of only 92. Furthermore, the yield of oil was only

8   83.7 weight %.